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A Study of Kapton Degradation Under Simulated Shuttle Environment

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I. INTRODUCTION

Weight loss and severe degradation of the surface of Kapton and other materials occur in low earth orbit. Atomic oxygen, the major ambient species at these altitudes and incident with approximately 5 eV energy in the ram direction, is the logical candidate for the cause of the observed degradation.

During the past three years we have developed a system which employs a source of low energy oxygen ions to simulate the shuttle low earth orbit environment. This source, together with diagnostic tools including surface analysis and mass spectroscopic capability, has been used to obtain quantum yields for atoms and molecules evolved from the surface of Kapton and other materials as the result of oxygen ion bombardment.

II. INSTRUMENTATION AND EXPERIMENTAL TECHNIQUE

Figure 1 shows the arrangement of the main components of our experimental apparatus within an ultra high vacuum system. The chemical composition of a surface can be determined by means of an Auger electron spectrometer. This can be done *in situ* before and after oxygen ion bombardment by simply rotating the sample holder (sample carousel) to move the sample from the position facing the ion gun to that facing the cylindrical mirror analyzer of the Auger electron spectrometer.

The low energy ion gun (developed by Kimball Physics, Inc., of Wilton, New Hampshire) produces a beam of oxygen ions with an energy variable from 500 eV to < 5 eV and an energy spread at this lowest energy of about 3 eV. The oxygen is admitted directly to the ionizer of the gun by way of a variable leak valve. For normal operating pressures of $\sim 10^{-1}$ Pa (10^{-3} Torr) within the gun, the O_2 pressure in the vacuum chamber is 5×10^{-5} Pa (4×10^{-7} Torr). The maximum ion current for sustained operation

of the gun is 0.1 μA, and the ion beam diameter at the target surface is ~ 2 mm. We have determined the composition of our ion beam in another vacuum chamber where the ion gun was positioned opposite a mass spectrometer. About 70% of the beam is O_2^+ and 30% O^+.

The quadrupole mass spectrometer, positioned so that its electron bombardment ionizer is only 3 cm from the surface being bombarded by the oxygen ions, detects the atoms and molecules evolved from the surface as a result of this bombardment. With the mass spectrometer set to detect a given mass number, the ion beam is gated on and off by a square wave voltage applied to the focus electrode of the gun, and the modulated component of the mass spectrometer output signal is synchronously detected with a lock-in amplifier.

Figure 2 shows the carbon monoxide (CO) signal (mass 28) from pyrolytic graphite for an ion energy of 93 eV. The left- and right-hand traces are, respectively, the amplitude (A) and quadrature (Asinϕ) outputs of the lock-in amplifier as functions of time recorded by a two-channel strip chart recorder. Each trace starts with a period of ~ 2 minutes during which the emission current of the electron bombardment ionizer of the gun is reduced to zero. This establishes a zero signal baseline from which the ion-induced signal can be measured. The emission is then turned up (the "on" portion of each trace) for about 3 minutes and then reduced again to zero to check for any baseline drift. During the on time, the graphite target is being bombarded by a 50 Hz square pulse ion current with a 50% duty cycle.

The off-on displacement in the A channel signal trace of Fig. 2 corresponds to an increase in CO pressure at the mass spectrometer of ~ 3×10^{-9} Pa(2×10^{-11} Torr). The background CO pressure in the vacuum

system at the time these traces were taken was 3×10^{-7} Pa (2×10^{-9} Torr). Without the use of lock-in detection techniques the ion bombardment induced CO signal would be lost in the "noise" (statistical fluctuations) of the signal arising from this background CO. Lock-in detection is useful even for atoms and molecules for which there is a negligible background pressure, since one can use the variation in amplitude (A) and phase (ϕ) with modulation frequency to extract information on the chemical kinetics involved in the surface interaction.[1]

III. RESULTS

We have measured the dependence on ion energy of the oxygen ion induced CO signals from pyrolytic graphite and Kapton.[2] For graphite the CO signal was examined at energies ranging from 4.5 to 465 eV and for Kapton from 4.5 to 188 eV. While the relative quantum yields inferred from the data are reasonably precise, there are large uncertainties in the absolute yields because of the assumptions necessary to convert our measured signal strengths to quantum yields. These assumptions are discussed in detail in Ref. 2. Our best present estimates are that the quantum yield (number of CO molecules evolved from the surface per incident oxygen ion) for graphite rises from 1.9 at 4.5 eV to 6.6 at 465 eV, and for Kapton varies from 1.6 at 4.5 eV to 5.1 at 188 eV. However, as discussed in Ref. 2, the energy with which the ions actually arrive at the Kapton surface is quite uncertain because of charging of the insulating Kapton surface by the oxygen ions and by electrons leaking from the ionizer of the mass spectrometer. We have considered possible systematic effects which would reduce these yields, and have concluded that it it unlikely that they are less than the given values by more than a factor of 4. While the above yields appear high, our Kapton data seems to fit nicely the curve

for reaction rate vs. ion energy inferred by Ferguson from mass loss data for Kapton from a number of investigations.[3]

The dependence of the amplitude and phase of our signals on the frequency with which the ion beam is modulated yields information on the chemical kinetics of the surface interaction.[1] Briefly stated, the surface reaction time leads to a decrease in A and an increase in the phase delay, ϕ, with increasing modulation frequency. The details of these variations in A and ϕ can be compared with the predictions of various models of the surface interaction to obtain reaction rates. We have obtained preliminary data for graphite and have submitted the following abstract to the 33rd National Symposium of the American Vacuum Society:

RATE CONSTANTS FOR THE SURFACE INTERACTION OF LOW ENERGY OXYGEN IONS WITH PYROLYTIC GRAPHITE* C.C. Horton, T.G. Eck, R.W. Hoffman, Department of Physics, Case Western Reserve University, Cleveland, Ohio, 44106.

The bombardment of pyrolytic graphite and Kapton surfaces by low energy oxygen ions results in the production of carbon monoxide (CO). Using a modulated ion beam, we have measured the magnitude and phase shift of the CO signal, detected by a quadrupole mass spectrometer, as a function of the modulation frequency of the ion beam. The data for pyrolytic graphite, for 93 eV ions incident on a room temperature surface and a modulation frequency varied from 30 to 2000 Hz, clearly indicates the presence of two components in the detected signal. This is characteristic of a "branched process" surface chemistry in which the reaction proceeds along multiple paths. Approximately 80% of the low frequency CO signal arises from a slow component with a rate constant of about 500 sec^{-1} (time constant of 2 msec). The remaining 20% of the signal is

associated with a process which has a rate constant at least ten times that of the slow component. Detailed data on pyrolytic graphite and data for Kapton will be presented.

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Data for the interaction of 93 eV oxygen ions with Nomex, Kevlar, and Teflon have been obtained as part of an investigation of the materials used in the construction of the tether for the first electrodynamic tethered satellite mission. For Nomex we see signals at mass numbers 14, 15, and 26 (which we attribute to N, NH, and CN) with quantum yields of ~ 0.5. For Kevlar the yields for these mass numbers are ~ 0.2. For Teflon we see a signal at mass 19 (attributed to F) with a yield of ~ 0.1 and one at mass 31 (attributed to CF) with a yield of ~ 0.4.

IV. PLANS FOR THE FUTURE

This work is continuing with the support of NASA Grant NAG 3-696. We plan to

(1) obtain detailed data for the dependence on modulation frequency of the CO signals from graphite and Kapton at 93 eV and 10 eV ion energy,

(2) look for and investigate signals from Kapton at other mass numbers,

(3) examine the effects of sample heating and ultra-violet irradiation on the ion-induced signals,

(4) extend our investigation of oxygen ion interaction with surfaces to refractory metals by looking for the production of volatile oxides of tungsten and molybdenum.

References

1. R. H. Jones, D. R. Olander, W. J. Siekhaus, and J. A. Schwarz, "Investigation of Gas-Solid Reactions by Modulated Molecular Beam Mass Spectrometry," J. Vac. Sci. Technol. 9, 1429 (1972).

2. C. C. Horton, T. G. Eck, and R. W. Hoffman, "Carbon Monoxide Production in Low Energy Oxygen Ion Bombardment of Pyrolytic Graphite and Kapton Surfaces," presented at the 32nd National Symposium of The American Vacuum Society and to be published in the Journal of Vacuum Science and Technology.

3. D. C. Ferguson, Proceedings of the 13th Space Simulation Conference, Oct. 1984, NASA CP-2340, p. 205-221.

Figure Captions

Figure 1. Schematic of apparatus: (A) ion gun, (B) mass spectrometer, (C) cylindrical mirror analyzer, (D) sample carousel.

Figure 2. Recorder traces for the CO signal from graphite for an ion energy of 93 eV, current of 0.1 μA, modulation frequency of 50 Hz, and lock-in time constant of 4 sec.

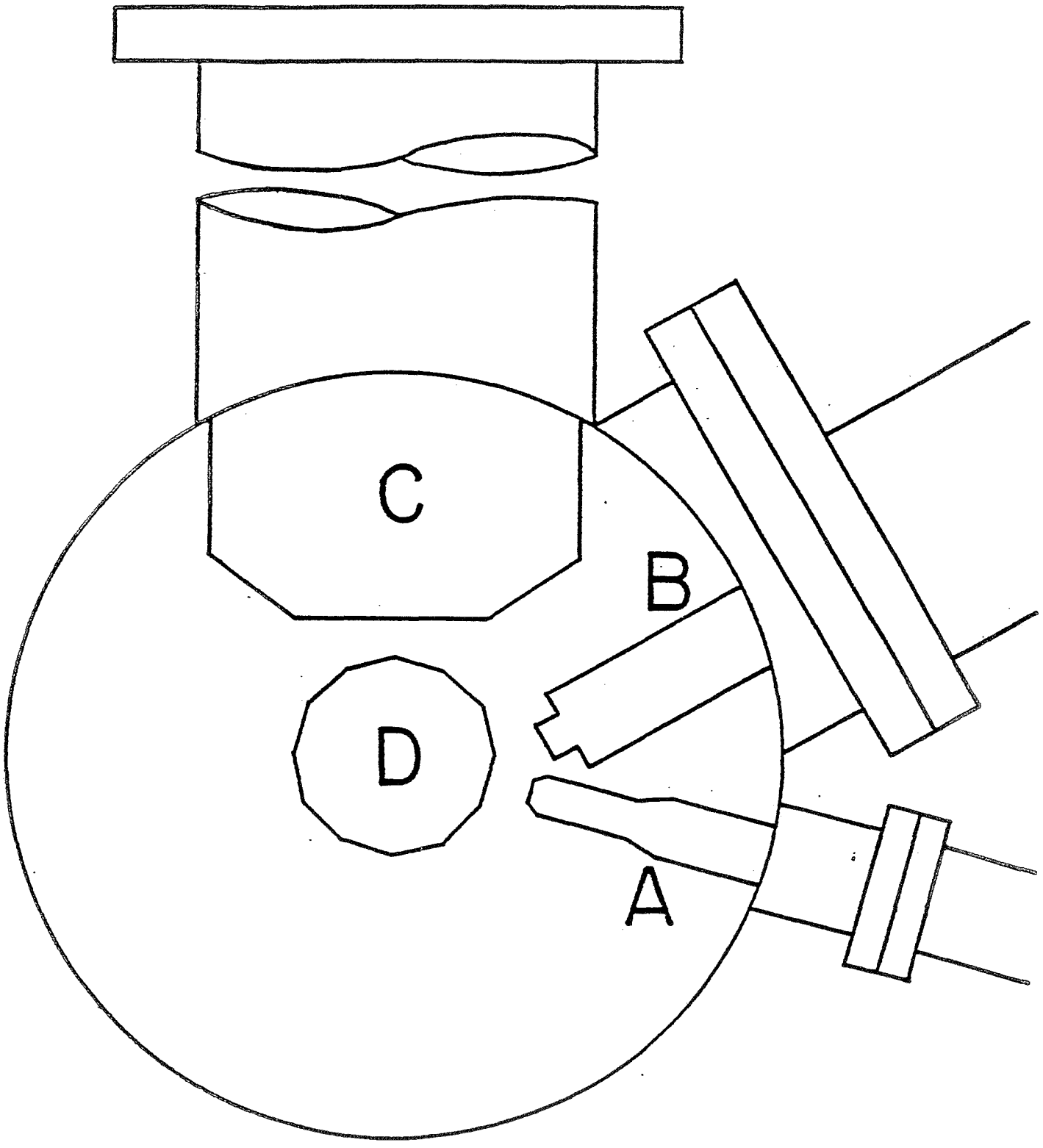

Fig. 1

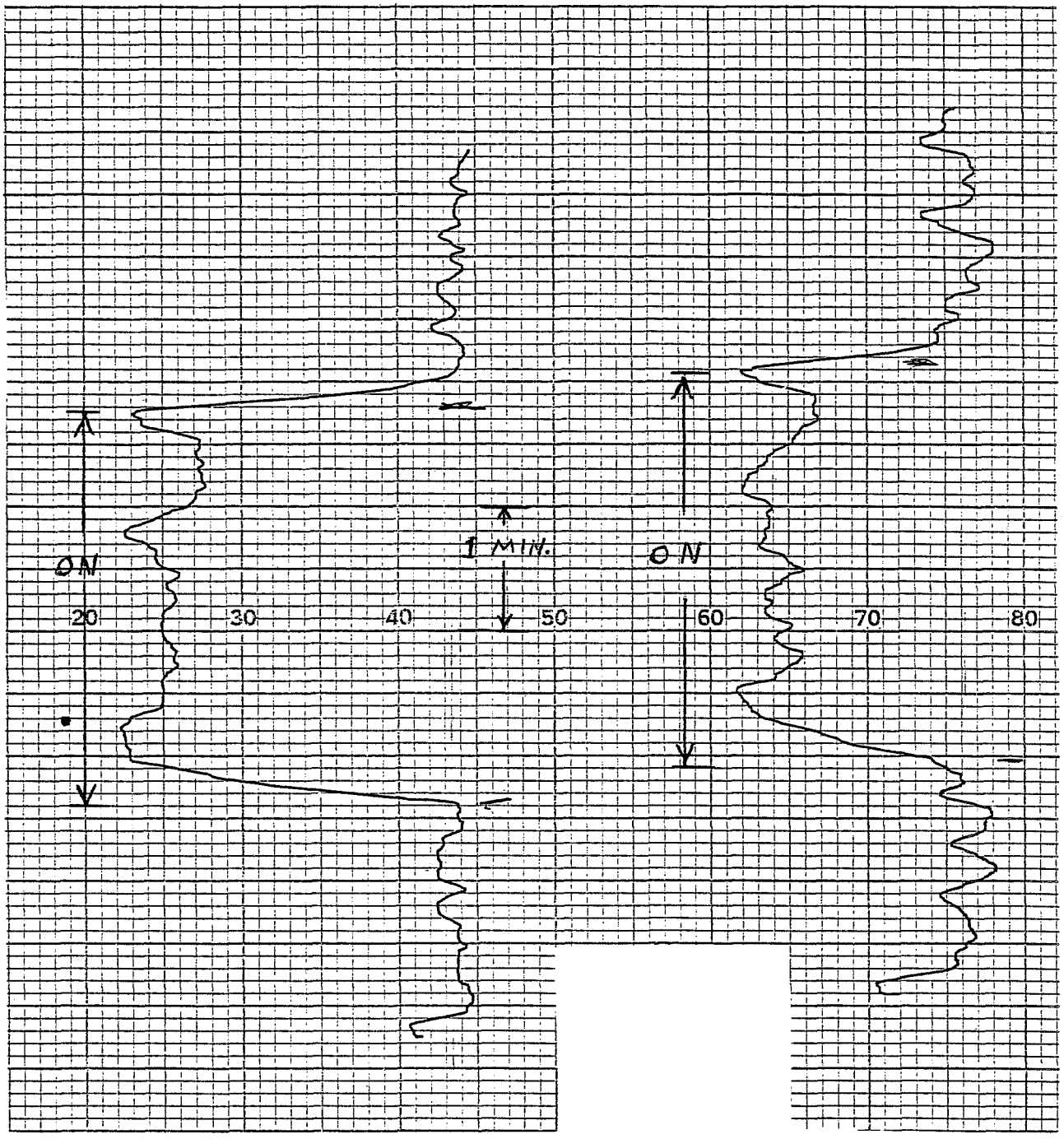

Fig. 2